

A two-stage surface treatment for the long-term stability of hydrophilic SU-8

Angela Sobiesierski,^{a*} Robert Thomas,^a Philip Buckle,^a David Barrow^b and Peter M. Smowton^a

The use of SU-8 photoresist as a structuring material for portable capillary-flow cytometry devices has been restricted by the near-hydrophobic nature of the SU-8 surface. In this work, we evaluate the use of chemical and plasma treatments to render the SU-8 surface hydrophilic and characterise the resulting surface utilising a combination of techniques including contact angle goniometry, atomic force microscopy and X-ray photoelectron spectroscopy. In particular, for low-power plasma treatments, we find that the chemistry of the plasma used to modify the SU-8 surface and the incorporation of O₂ on that modified surface are paramount for improved surface wettability, whilst plasma-induced surface roughness is not a necessary requirement. We demonstrate a technique to obtain a hydrophilic SU-8 surface with contact angle as low as 7° whilst controlling and significantly reducing the level of surface roughness generated via the applied plasma. An additional chemical treatment step is found to be essential to stabilise the activated SU-8 surface, and incubation of the samples with ethanolamine is demonstrated as an effective second-stage treatment. Application of the optimised two-stage surface treatment to cross-linked SU-8 is shown to result in a smooth hydrophilic surface that remains stable for over 3 months. Copyright © 2015 The Authors *Surface and Interface Analysis* Published by John Wiley & Sons Ltd.

Keywords: SU-8; hydrophilic surface; contact angle; XPS

Introduction

SU-8 is an epoxy resin with a monomer composed of, on average, eight aromatic benzene rings and eight cyclic ether epoxide rings. With the addition of a photo-acid generator and solvent, SU-8 can be spun on with thicknesses ranging from sub-micron to hundreds of microns, giving rise to its widespread use as a structuring material within microelectromechanical systems applications.^[1–3] SU-8 is compatible with standard GaAs-based semiconductor device fabrication techniques^[4] and is thus directly applicable to opto-fluidic lab-on-a-chip type devices where it can be used to construct three-dimensional channels within which analytes of interest can flow between closely integrated semiconductor devices such as laser diodes and detectors. The known high chemical resistance of cross-linked SU-8,^[5] combined with its transparency across the visible part of the optical spectrum enables it to provide the necessary physical separation of the analyte from the surrounding potentially harmful or bio-fouling GaAs-based semiconductor materials without itself causing any chemical or optical interference.

These material properties combined with the biocompatibility of SU-8^[6] facilitates its application to the area of bio-health. However, the surface of SU-8 is insufficiently hydrophilic in its untreated state to enable lab-on-a-chip flow cytometry as described earlier without involving the use of external pumps and flow circuitry. This would limit its use in the area where its application would see most benefit – in portable, self-sufficient devices that provide point of care analysis and diagnostics in resource poor locations.

Several approaches exist to increase the hydrophilicity of the SU-8 surface. A key parameter used to evaluate the effectiveness of the treatment applied is the contact angle, θ . This is defined as the angle where a liquid/vapour interface meets a solid surface (Fig. 1). The wettability of a surface can be determined from the force

balance relation where γ is defined as the interfacial free energy between the different solid, liquid and vapour phases. At thermal equilibrium, $\gamma_{sv} = \gamma_{sl} + \gamma_{lv} \cos \theta$. The degree of wetting or wettability of a surface is thus directly related to the contact angle θ with a lower contact angle indicative of a more hydrophilic surface.

Application of acid-based treatments such as acetic acid with the addition of ceric ammonium nitrate (CAN) has been shown,^[7] via contact angle measurements, to result in increased wettability of the SU-8 surface. The cerium within the CAN is believed to act as a catalyst in opening the epoxide rings present on the SU-8 surface. An alternative less chemically aggressive technique makes use of an oxygen plasma^[8] to modify the functional groups on the surface of the SU-8. This has been reported to reduce the contact angle of a droplet of deionised (DI) water on the treated SU-8 surface from over 80° to below 20°. However, depending on the power applied, this treatment has been found to result in a significantly roughened surface. The importance of plasma-induced surface roughness as a requirement for enhanced surface wettability is not well known.

In this work, we apply both CAN and low-power plasma treatments to SU-8 to obtain a hydrophilic surface. We employ contact angle goniometry to measure the surface wettability, atomic force

* Correspondence to: A. Sobiesierski, School of Physics and Astronomy, Cardiff University, 5 The Parade, Cardiff, CF24 3AA, UK.
E-mail: kestlea@cf.ac.uk

This is an open access article under the terms of the Creative Commons Attribution License, which permits use, distribution and reproduction in any medium, provided the original work is properly cited.

a School of Physics and Astronomy, Cardiff University, 5 The Parade, Cardiff, CF24 3AA, UK

b School of Engineering, Cardiff University, 5 The Parade, Cardiff, CF24 3AA, UK

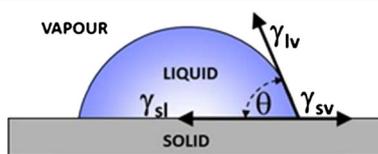


Figure 1. Interfacial free-energy force diagram for a droplet of liquid on solid surface. θ is the contact angle that is used as a measure of surface wettability, and γ is the interfacial free energy between the different solid, liquid and vapour phases (after Romé-hart^[16]).

microscopy (AFM) to determine surface topographical changes and X-ray photoelectron spectroscopy (XPS) to gain insight into the chemical changes occurring at the SU-8 surface for both treatment methods.

Furthermore, by using alternative gases in addition to O_2 for the plasma treatment, we can separate out the physical and chemical reactions occurring at the surface and explore the connection between surface wettability of SU-8 and plasma-induced surface roughness. From this, we are able to identify the main process parameter required to achieve a hydrophilic SU-8 surface by plasma treatment, we establish the role of plasma-induced surface roughness and we demonstrate a smooth hydrophilic SU-8 surface using a low-power O_2 plasma treatment.

The second part of this work focuses on the long-term stability of the hydrophilic SU-8 surface. Previous studies^[9] have reported that in general the effect of a single stage of hydrophilic treatment is temporary, with the SU-8 surface reverting back to hydrophobic. In order to provide a working shelf life of 3 months or more for portable flow cytometry devices reliant on a hydrophilic SU-8 surface, a second-stage treatment is required that bonds to the functional groups generated on the SU-8 surface by the first stage of treatment and results in a stable hydrophilic surface layer.

Reports in the literature of techniques to preserve the hydrophilic SU-8 surface have included covalent bonding of polyethylene glycol^[10] and bio-grafting or photo-grafting of hydrophilic polymers^[11] or hydrogels^[12] onto the modified SU-8 surface. However, these techniques involve chemical procedures that can be hazardous, requiring the SU-8 samples to be initially immersed in concentrated sulfuric acid at 85 or 90 °C, or complex – subjecting the SU-8 samples to a series of photosensitive chemicals, UV exposures and overnight soaks in solutions, often under a N_2 atmosphere.

Historically, epoxy resin has been cured by homopolymerisation or by reaction with amines, anhydrides or phenols that act as curing agents or hardeners. Out of these, the most promising for application to SU-8 is the amine group as it does not require any significant application of heat for the reaction to occur. In particular, primary amines are extremely effective as a chain reaction can occur where the NH_2 group of the amine initially reacting with the epoxide forms a hydroxyl group whilst also generating a secondary amine that can then react still further to generate a tertiary amine and additional hydroxyl group. This reaction, which should continue until all opened epoxide groups are saturated, should therefore both stabilise the epoxide and also provide additional hydroxyl groups on the surface.

Ethanolamine is a primary amine, and a primary alcohol (due to a hydroxyl group), and has previously been shown^[7] to increase the surface wettability of SU-8 surfaces following a first CAN treatment step. It is a simple treatment that is applicable to integrated optical devices without causing any degradation to active optical components. To our knowledge, it has not been applied before to a hydrophilic SU-8 surface generated by a plasma treatment. To determine the effectiveness of ethanolamine as a stabilising material, the results of our first-stage treatment studies were used to

generate a series of hydrophilic SU-8 samples utilising both CAN and O_2 plasma treatment methods. The activated SU-8 surfaces were then incubated with ethanolamine. A schematic diagram to illustrate the chemical bonds that are formed during these treatment steps is provided in Fig. 2. To determine the long-term effectiveness of the two-stage treatment, the stability of the SU-8 was monitored, using contact angle goniometry, for a period of time in excess of 3 months. Furthermore, a subset of samples was kept under N_2 ambient to determine any benefit or otherwise of keeping the treated SU-8 surfaces under an inert atmosphere.

Experimental section

Reagents

SU-8 2075 was purchased from Oxford Instruments Plasma Technology, Bristol, UK Microchem (Durham, UK). Chrome Etch MS8 was purchased from Chetech (Rugby, UK). Ethanolamine and sodium phosphate were purchased from Sigma-Aldrich (Poole, UK). Acetone and isopropanol were purchased from Fisher Chemicals (Loughborough, UK).

Sample preparation

Preliminary sample preparation

SU-8 2075 was spin-coated onto glass coverslips that had previously been cleaned with acetone and isopropanol and dehydrated

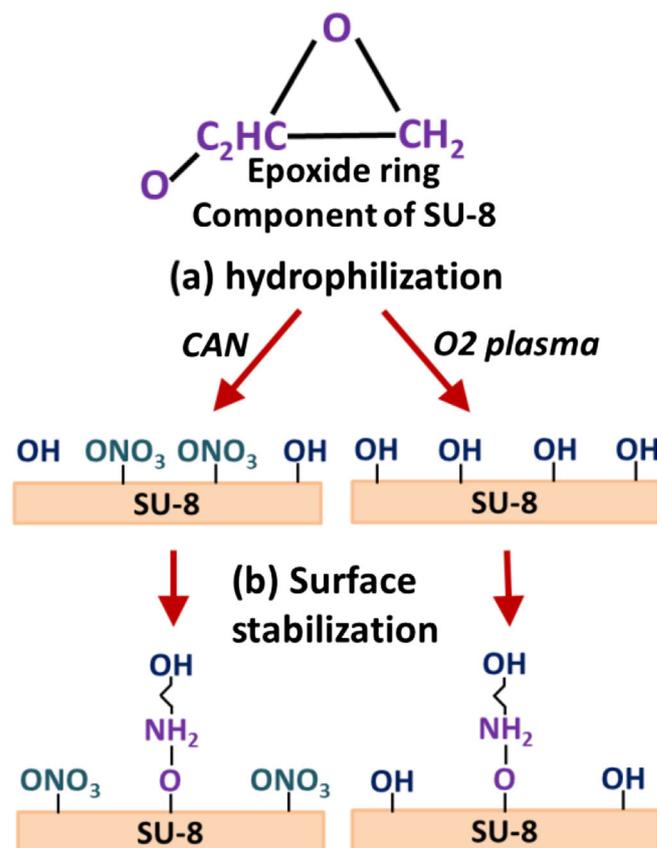


Figure 2. Schematic of chemical modifications to the SU-8 surface following the two stages of hydrophilic treatment for both CAN and O_2 plasma first-stage treatment.

at 180 °C for 2 min minimum. A spin speed of 8000 rpm and an acceleration of 300 rpm/s for 45 s were used. The samples were soft baked on hotplates at 67 °C for 2 min and 97 °C for 6 min, respectively. The samples were then blanket exposed to UV light using a Karl Suss MJB3 mask aligner for 30 s with an intensity of 20 mW/cm² at 405 nm. A post-exposure bake of 67 °C for 2 min and 97 °C for 6 min was subsequently applied followed by a final hard bake at 180 °C for 20 min. This resulted in fully cross-linked SU-8 layers of approximately 50 μm thick.

First-stage hydrophilic treatment

1. Plasma treatment

A low-power gas plasma was applied to the samples using an Oxford Instruments Plasma Technology inductively coupled plasma (ICP) 100 system (Bristol, UK). For entry into the ICP chamber, the samples were placed, unclamped, on a 4-in. silicon carrier wafer. A typical plasma recipe consisted of 40 sccms of gas at a chamber pressure of 10 mTorr, reactive-ion etching power of 50 W and ICP power of 300 W. The lower electrode temperature was maintained at 25 °C, using liquid nitrogen cooling. In addition He cooling was applied to the back side of the silicon carrier wafer to minimise plasma-induced heating of the samples.

2. Acid treatment

MS8 Chrome etchant that consists of CAN (25–30%) and acetic acid (5–10%) was maintained at 50 °C on a hotplate located within a fume cupboard. The samples were immersed in the etchant for 1 h maximum and then rinsed, with agitation, for a minimum of 5 min using 15 MΩ DI water.

Second-stage surface stabilisation treatment

A 0.1-M solution of ethanolamine was prepared in a sodium phosphate buffer. The solution was warmed to 50 °C, and the samples were incubated for up to 1 h. The samples were then rinsed, with agitation, in a solution of sodium phosphate for a minimum of 5 min.

Characterisation techniques employed

Contact angle measurements

Static sessile drop contact angle measurements were made using a goniometer with a high-resolution digital camera. For each sample, the contact angle was measured a minimum of four times using a DI water droplet size of 0.6 μl, and the average value recorded. The maximum deviation from the average value was used to determine the error in the contact angle measurements and was estimated to be ±2.5°. All measurements were conducted within a class 1000 clean room where the room temperature was maintained at 19–20 °C. The relative humidity was monitored throughout with values in the range of 33–60% being recorded.

Surface roughness measurements

To determine the effect on surface topography of the different surface treatments applied, a NanoScope IIIa Multimode (Digital Instruments now Bruker, Coventry, UK) AFM was employed, operating in tapping mode, under ambient conditions and with an n-type Si cantilever.

XPS photoelectron spectroscopy

The chemical modifications made to the SU-8 surface following both acid and plasma treatments were assessed by an XPS analysis. A Kratos Axis Ultra DLD system (Manchester, UK) was used to collect the XPS spectra using a monochromatic Al Kα X-ray source operating at 120 W. Data were collected with pass energies of 160 eV for the survey spectra, and 40 eV for the high-resolution scans. The system was operated in the hybrid mode, utilising a combination of magnetic immersion and electrostatic lenses, and the data were acquired over an area approximately 300 × 700 μm². A magnetically confined charge compensation system was used to minimise charging of the sample surface, and all spectra were taken with a 90° take off angle. A base pressure of approximately 1 × 10⁻⁹ Torr was maintained during collection of the spectra. Spectra were calibrated to the C(1s) line, taken to be 285.0 eV as appropriate for organic species. Quantification of the elemental ratios was performed using CasaXPS v2.3.17, after subtraction of a Shirley background, and sensitivity factors were supplied by the manufacturer.

Results and discussion

Initial treatment studies

SU-8 2075 samples were prepared on glass coverslips and subjected to either a CAN chemical or O₂ plasma treatment. For the CAN treatment step, we found that a 1-h treatment was sufficient to reduce the contact angle from 84° to 11° with a root-mean-square (rms) surface roughness of 1 nm. Extended treatment times, as reported elsewhere,^[7] were found for our samples to result in complete delamination of the SU-8 layer from the glass with some evidence of swelling of the SU-8. A 2-min low-power O₂ plasma resulted in a contact angle of 19° but with an rms surface roughness of 8 nm.

Contact angle versus surface roughness

To investigate the relationship between plasma-induced surface roughness and surface wettability, we apply different gas plasmas independently to SU-8 samples, using an ICP etching tool. In addition to O₂, the gas species chosen for this study are Ar and C₄F₈:O₂ in a 0.95:0.05 ratio. An Ar plasma is routinely used to clean sample surfaces by physical bombard and should result in a roughened surface but with no chemical reaction occurring. In contrast, a small fraction of fluoride gas added to O₂ should result in a very smooth chemically etched SU-8 surface.^[13]

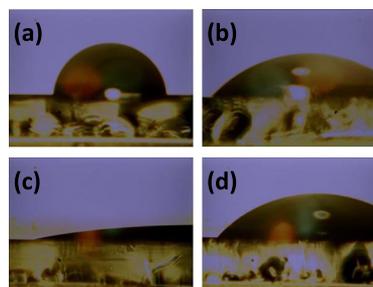


Figure 3. Droplet size of 0.6 μl of DI water on (a) untreated SU-8 and (b) Ar, (c) O₂ and (d) C₄F₈/O₂ plasma treated surfaces.

Figure 3 shows 0.6 μl droplets of DI water on (a) untreated SU-8 and after 2 min plasma treatments with (b) Ar, (c) O_2 and (d) $\text{C}_4\text{F}_8/\text{O}_2$, respectively. For untreated SU-8, the contact angle was measured to be 84° . Out of the three gas plasmas applied the lowest contact angle measured, 19° was for the sample subjected to an O_2 plasma. For the samples that received Ar and $\text{C}_4\text{F}_8/\text{O}_2$ plasma treatments, the contact angles were measured to be 32° and 46° , respectively. Therefore, of the three gas plasmas applied, O_2 is the most effective hydrophilisation treatment. When the duration of the plasma treatment was increased to 4 and 8 min, samples subjected to O_2 plasma resulted in contact angles of 15° and 7.5° , respectively. In comparison, samples subjected to Ar and $\text{C}_4\text{F}_8/\text{O}_2$ showed no further reduction in contact angle.

Atomic force microscopy analysis of the resulting sample surfaces shows (see Supporting information for images) that application of an O_2 plasma to SU-8 causes the largest increase in surface roughness. rms roughness for a 2-min O_2 plasma was measured to be 8 nm. In comparison, the rms roughness for both Ar and $\text{C}_4\text{F}_8/\text{O}_2$ 2-min plasmas remained much closer to that of untreated SU-8, at 0.23 nm. Table 1 provides a summary of contact angle and rms roughness measurements recorded for all treatments applied.

It is known^[8] that Sb accumulates on the surface of SU-8 when subjected to an O_2 plasma. This is believed to come from the photo-acid generator, triarylsulfonium/hexafluoroantimonate contained within the SU-8. By adding a small fraction of a fluoride gas to the plasma, the Sb can be removed *in situ*.^[13] However, as shown earlier, we have found that this results in a far less effective hydrophilisation treatment than O_2 . It has also been shown^[8] that it is possible to remove the Sb post-etch by simply rinsing the sample in DI water, with agitation, in an ultrasonic bath.

Without removal, we believe that the Sb may act as a micro-mask that causes the plasma treatment to become non-uniform across the sample surface. As the plasma duration is increased, additional Sb masking causes the surface to become more uneven and rough. To verify this, further O_2 plasma treatments were applied to SU-8 samples with identical ICP etch process parameters as before and for total etch times of 2, 4 and 8 min. This time, however, the plasma treatment was split into 2-min steps: following each 2-min plasma, the samples were removed from the ICP and rinsed in DI water using an ultrasonic bath before continuing with the next 2-min plasma. A comparison of the resulting contact angle and surface roughness measurements is shown in Fig. 4.

The single plasma treatment (solid blue circles) shows a direct relation between plasma duration and surface roughness. By splitting the plasma into shorter steps (solid red squares) and applying the water rinses, we are able to significantly reduce the plasma-induced

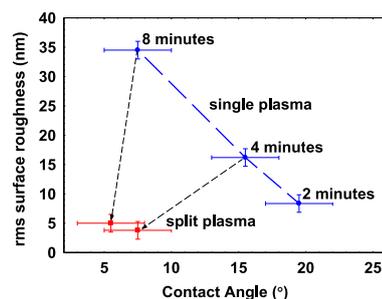


Figure 4. Comparison of rms surfaces roughness with contact angles measured for single (blue) and split (red) O_2 plasma treatments. The split treatment consists of a multiple number of 2-min plasma treatments followed by a DI water rinse.

surface roughening. The contact angles following this treatment were measured to be 7° and 5° for two and four 2-min plasma steps, and the surface roughness was measured to be 3 and 5 nm, respectively. Removing the Sb before proceeding with each further 2-min plasma treatment therefore resulted in a more uniform surface treatment and smoother surface whilst also achieving a large reduction in contact angle – comparable with that obtained for an 8-min single O_2 treatment.

This demonstrates that over the range of surface roughness measured in this study for plasma treated SU-8 surfaces, i.e. 0.3 to 32 nm, plasma-induced surface roughness is not a requirement for improved wettability of the treated surface – the key to achieving a hydrophilic surface is the chemistry of the plasma used to modify the SU-8 surface and the incorporation of O_2 on that modified surface.

Whilst the use of an Ar plasma did reduce the measured contact angle to some extent, the effect was limited by the absence of O_2 during the treatment step. The reduction in effectiveness of the hydrophilic treatment when 5% of the O_2 gas was replaced with C_4F_8 was because the addition of fluoride caused the surface material to be removed by chemical etch rather than being chemically modified.

Surface chemistry of treated surfaces

The results of the studies detailed in the previous sections confirm that the optimum plasma treatment for SU-8 requires the inclusion of O_2 and that CAN is also an effective chemical treatment. We now focus on understanding the chemical modifications occurring on the SU-8 surface for both techniques.

Table 1. Contact angle and surface roughness measurements for different surface treatments applied to SU-8 samples

| Treatment | Duration (min) | Contact angle (°) | Surface roughness, rms (nm) | |
|-----------|-----------------------------------|-------------------|-----------------------------|-----|
| Control | – | 84 | 0.23 | |
| Plasma | Ar | 32 | 0.6 | |
| | $\text{C}_4\text{F}_8/\text{O}_2$ | 46 | 0.3 | |
| | O_2 | 19 | 8.0 | |
| | O_2 | 15 | 16.0 | |
| | O_2 | 7 | 32.0 | |
| | O_2 split + DI rinse | 2 + 2 | 7 | 3.0 |
| | O_2 split + DI rinse | 2 + 2 + 2 + 2 | 5 | 5.0 |
| Chemical | CAN | 11 | 1.0 | |

rms, root mean square; DI, deionised; CAN, ceric ammonium nitrate

The full survey XPS spectra for both O₂ plasma and CAN surface treatments are shown in Fig. 5. It reveals some significant differences between the samples.

For the O₂ plasma treated sample, a large amount of Sb was confirmed to be on the surface. Following a 5-min DI water rinse as described in the previous section, a further XPS scan confirmed that the level of Sb recorded for a rinsed sample had reduced to that for an untreated sample. In comparison, the CAN treatment resulted in a large amount of cerium remaining on the surface that could not be removed with a water rinse. This may have implications for bio-health applications.

To determine how the SU-8 surface has been chemically modified by the different treatments, detailed XPS of the C 1s spectra was obtained. In Fig. 6, the peaks of the untreated SU-8 sample have been identified^[14,15] as aromatic carbon (C–C) at 285 eV and C–O at 286.6 eV. A small peak is also observed at 291.3 eV that is shake-up related to the benzene rings. Comparison of the spectra indicates that for both the O₂ plasma and CAN treatments, there is a reduction in the C–C signal, because of accumulation on the surface of chemical species including cerium, antimony, oxygen and hydroxyl groups, and a relative increase in both C–OH (shoulder on main C–C peak) and, in particular for the CAN treated sample, carboxylic acid COOH[–] at 289.3 eV. The presence of OH containing functional groups on the treated SU-8 surfaces indicates that the O₂ plasma and CAN treatments are both effective techniques for generating a hydrophilic SU-8 surface.

Stability of hydrophilic surface

Figure 7 shows the reversal of the contact angle with time for SU-8 samples subjected to O₂ plasma and CAN treatments.

It can be seen that the reversal rate is quite different for the two treatments applied. Whilst the effect of both treatments is to open

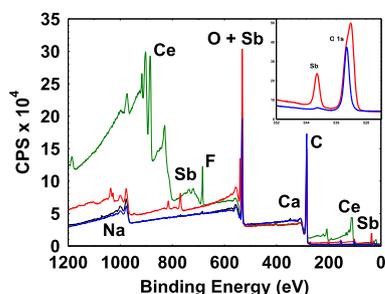


Figure 5. Full survey spectra for untreated SU-8 (blue), O₂ plasma (red), O₂ plasma + DI water rinse (black) and CAN (green). Inset shows detail of the O 1s and Sb 3d peaks for the untreated and O₂ plasma treated surfaces.

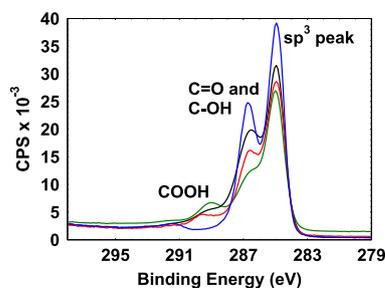


Figure 6. C1s XPS spectra for untreated SU-8 (blue), O₂ plasma (red), O₂ plasma + DI water rinse (black) and CAN (green).

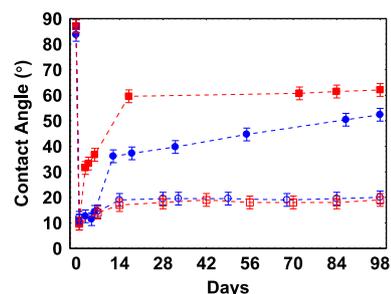


Figure 7. Measured contact angles as a function of time for O₂ plasma (solid blue circles) and CAN (solid red squares) treatments. Also shown (clear circles and squares) are the contact angles measured for the same treatments plus incubation of the samples with ethanolamine.

epoxide rings on the SU-8 surface, the chemistry involved in the surface reactions is different. For example, a significant amount of cerium is present in the CAN treatment but absent from the O₂ plasma treatment. It may be that the surface compositions generated by the different treatments causes a variation in the stability of the hydrophilic surface. The behaviour of the two treatments is also different. For example, repeated treatment steps with O₂ plasma on the same sample results in a decreasing contact angle down to 5°, whereas with repeated CAN treatments, the resulting contact angle appears to reach a minimum at approximately 10°, even with replenished CAN solution. One hypothesis is that the O₂ plasma simply opens more epoxide rings on the SU-8 surface than the CAN treatment. This may be due to the bombarding nature of the plasma treatment being more effective than a chemical treatment on what is known to be a surface with high chemical resistance.

The additional opened epoxide rings caused by the plasma treatment may then prolong the hydrophilic nature of the SU-8 surface. The use of high power plasma treatment as a means to slow the reversal of the hydrophilic SU-8 surface back to hydrophobic was investigated by Walther,^[9] but the high powers used resulted in large plasma-induced surface roughening, of the order of 200 nm.

For both treatments considered here, the contact angle increases, within 30 days, to typically 40°. Therefore, although the time of reversion to a near-hydrophobic surface is significantly shorter for the CAN treatment compared with the O₂ plasma, the reduction in wettability for both treatment methods over such a relatively short time scale results in an SU-8 surface insufficiently hydrophilic for fluid flow by capillary action and confirms the requirement for a second-stage treatment to maintain a hydrophilic SU-8 surface.

The stability of SU-8 samples that have received either the CAN or O₂ plasma treatment plus a secondary ethanolamine treatment step is also shown in Fig. 7. The treatment with ethanolamine results in an additional slight improvement in surface wettability with a reduction in contact angle to approximately 5°. This is believed to be due to the free hydroxyls provided by the ethanolamine. However, within a day, this had reverted back to a small increase in contact angle to approximately 19°. However, no further increases in contact angle were measured for the SU-8 samples over a time period in excess of 3 months confirming that the SU-8 hydrophilic surface had been stabilised by the adsorption of ethanolamine. No significant difference in contact angle measurements was found for the subset of samples kept under N₂ indicating that there was no benefit of keeping the samples under an inert atmosphere.

Within this study, we also included an SU-8 sample that did not receive the first hydrophilisation stage of treatment but was subjected to the ethanolamine step. For fully cross-linked SU-8 without a first treatment step to open the epoxide rings on the surface, there should be far fewer epoxide rings available for the ethanolamine to react with. The resulting contact angle measured for this sample was 74° – only a slight increase in surface wettability from untreated SU-8 (84°). This confirms the importance of applying both the surface activation and surface stabilisation steps to obtain a stable long-lasting hydrophilic SU-8 surface with maximised surface wettability.

Conclusion

We have investigated the physical and chemical modifications caused to SU-8 surfaces by the application of various hydrophilisation treatments. Whilst we find that the inclusion of O₂ on the surface is key, the roughness induced by an O₂ plasma is not found to be a necessary condition to achieve a significant improvement in surface wettability and can be reduced and/or controlled by applying the treatment in steps separated by water rinses. In particular, both CAN and O₂ plasma treatments prove effective at generating a hydrophilic SU-8 surface with contact angles as low as 7° being measured. However, with no further treatment, the hydrophilicity of the SU-8 surface reverts back towards the native, untreated state within a matter of days (CAN) or weeks (O₂ plasma). The application of an ethanolamine incubation treatment to the activated SU-8 surface ensures that the surface remains hydrophilic with a contact angle of approximately 19° for a time period in excess of 3 months. Regarding the long-term stability of the hydrophilic SU-8 surface, no benefit was seen of storing the samples under an inert atmosphere.

Acknowledgements

This work was supported by EPSRC grant EP/L005409/1. Information on how to access all data supporting the results in this article can be found at Cardiff University data catalogue at <http://dx.doi.org/10.17035/d.2015.100123>. We thank Professor Philip Davies and Dr David Morgan for help with the XPS measurements and Dr Andriy Moskalenko for help with the AFM measurements.

References

- [1] E. Conradie, D. Moore, SU-8 thick photoresist processing as a functional material for MEMS applications, *J. Micromech. Microeng.* **2002**, *12*, 368–374.
- [2] C. Liu, Recent developments in polymer MEMS, *Adv. Mater.* **2007**, *19*, 3783–3790.
- [3] J. Zhang, K. L. Tan, G. D. Hong, Polymerization optimization of SU-8 photoresist and its applications in microfluidic systems and MEMS, *J. Micromech. Microeng.* **2001**, *11*(1), 20–26.
- [4] S. Cran-McGreehin, K. Dholakia, T. Krauss, Monolithic integration of microfluidic channels and semiconductor lasers, *Opt. Express* **2006**, *14*, 7723–7729.
- [5] H. Lorentz, M. Despont, N. Fahrni, N. LaBlance, P. Renaud, SU-8: a low cost negative resist for MEMS, *J. Micromech. Microeng.* **1997**, *7*, 121–124.
- [6] K. V. Nemanja, K. L. Moodie, J. B. Brennick, A. Sud, B. Gimia, *In vitro* and *in vivo* evaluation of SU-8 biocompatibility, *Mater. Sci. Eng., C.* **2013**, *33*, 4453–4459.
- [7] M. Nordström, R. Marie, M. Calleja, A. Boisen, Rendering SU-8 hydrophilic to facilitate use in micro channel fabrication, *J. Micromech. Microeng.* **2004**, *14*, 1614–1617.
- [8] F. Walther, T. Drobek, A. Gigger, M. Hennemeyer, M. Kaiser, H. Herberg, T. Shimitsu, G. Mordill, R. Stark, Surface hydrophilization of SU-8 by plasma and wet chemical processes, *Surf. Interface Anal.* **2010**, *42*, 1735–1744.
- [9] F. Walther, P. Davydovskaya, S. Zürcher, M. Kaiser, H. Herberg, A. Gigger, R. Stark, Stability of the hydrophilic behaviour of oxygen plasma activated SU-8, *J. Micromech. Microeng.* **2007**, *17*, 524–531.
- [10] C. Choi, I. Hwang, Y.-L. Cho, S. Han, D. Jo, D. Jung, D. Moon, E. Kim, C. Jeon, J. Kim, T. Chung, T. Lee, Fabrication and characterisation of plasma-polymerized poly(ethylene glycol) film with superior biocompatibility, *Appl. Mat. and Int.* **2013**, *5*, 697–702.
- [11] C.-L. Wu, M.-H. Chen, F.-G. Tseng, SU-8 hydrophilic modification by forming copolymer with hydrophilic epoxy molecule. *7th International Conference on Miniaturized Chemical and Biochemical Analysts Systems, California, USA*. October 5–9, **2003**.
- [12] Z. Gao, D. Henthorn, C.-S. Kim, Enhanced wettability of an SU-8 photoresist through a photografting procedure for bioanalytical device applications, *J. Micromech. Microeng.* **2008**, *18*, 045013.
- [13] K. Rasmussen, S. Keller, F. Jensen, A. Jorgensen, O. Hansen, SU-8 etching in inductively coupled oxygen plasma, *Microelectron Eng.* **2013**, *112*, 35–40.
- [14] T. Takahagi, A. Ishitani, XPS studies by use of the digital difference spectrum technique of functional groups on the surface of carbon fiber, *Carbon* **1984**, *22*, 43–46.
- [15] G. Beamson, D. Briggs, High resolution XPS of organic polymers: the Scienta ESCA300 database, *J. Chem. Edu.* **1993**, *70*, A25.
- [16] *ramehart instrument co.* **2015**; <http://www.ramehart.com/contactangle.htm>